Addendum To The Holding Time Study For Environmental Samples In Frozen Archive

For the Chemistry Working Group of the
Mississippi Canyon 252 Trustees

Version 3.0
November 25, 2013

ADDENDUM TO THE HOLDING TIME STUDY FOR ENVIRONMENTAL SAMPLES IN FROZEN ARCHIVE

Approval of this analytical addendum is for the purposes of obtaining data for the Natural Resource Damage Assessment (NRDA). Each party reserves its rights to produce its own independent interpretations and analyses of any data collected pursuant to this work plan.

This analytical addendum will be implemented consistent with existing trustee regulations and policies. All applicable state and federal permits will be obtained prior to conducting work.

APPROVED:

_____ _____

NOAA Trustee Representative: Date

_____ _____

Louisiana Trustee Representative: Date

_____ _____

BP Representative: Date

Study Plan for NRDA

Frozen Sample Chemical Stability: Laboratory Analysis Plan
Addendum 1

Chemistry Technical Working Group

Version 3.0, November 25, 2013

1.0. Purpose:

The original Holding Time Study Laboratory Analysis Plan (version 2.0, August 2011) evaluated the stability of fresh-frozen tissues collected as part of the NOAA National Status and Trends Mussel Watch program. Mussel Watch samples were selected because of the objective to re-test samples that had been properly archived for several years; at that time the earliest samples collected under the DWH NRDA were less than two years old. The findings from this evaluation, along with other evidence in published and unpublished literature, led to amendment of the holding times for extraction of samples for hydrocarbons in tissues and sediments in the Mississippi Canyon 252 (Deepwater Horizon) Analytical Quality Assurance Plan (AQAP) from 1-year to 4-years (AQAP, version 3.1). The purpose of this addendum is to further evaluate the stability of hydrocarbons, this time in selected frozen sediment/soil samples which were collected and analyzed during the summer and fall of 2010. This additional testing will be used to supplement the data and evaluation performed in the original work plan with samples collected as part of the Deepwater Horizon NRDA analyzed for a broader suite of contaminants.

2.0 Project Scope:

Five sediments samples and one soil sample will each be extracted in triplicate (total number of sample analyses is 18 = 6 x 3) at Alpha Analytical Laboratory in Mansfield, MA (Alpha). These samples were received at Alpha within two days of collection in 2010 and have been maintained frozen at -20 °C, except for the time during which the samples were originally analyzed. The list of proposed samples is shown in Table 1 at the end of the addendum. These samples were chosen based on:

- Dates of collection (collected early in the NRDA process);
- Concentrations of alkylated PAHs (they contain PAHs at levels above the method detection limit allowing for accurate comparison between initial and re-extraction results and cover a broad range of concentrations);
- Petrogenic PAH profiles similar in complexity to fresh or slightly weathered oil,
- Volume of sample available for analysis; and
- Matrices (sediments and soil)

The samples will be analyzed for the alkylated PAHs, saturated hydrocarbons/ total extractable hydrocarbons, petroleum biomarkers, and percent moisture using the same preparation, extraction, cleanup, and analysis techniques employed by Alpha during the initial extraction/analysis of these samples. Quality Assurance and Quality Control (QA/QC) criteria as specified in Table 6.1 of the

AQAP will be followed (e.g., each analytical batch of up to 20 field samples will contain a method blank, matrix spike, and SRM 1941b as batch quality control and MC252 Reference Oil will be analyzed at the instrument per the AQAP).

3.0 Objectives:

- Analyze three replicates of the six sediment and soil samples listed in Table 3 below (Tables 1 & 2 are in the original work plan). This testing is an expansion of empirical data gathered under the initial effort and used to support the holding times for frozen sediment, tissue, filters, and inert sorbent material in Table 3.1 of the AQAP.
- Assess long-term stability of sediment and soil samples collected during the Deepwater Horizon NRDA through statistical comparison of original data with the new data produced through execution of this addendum. Relative Percent Difference (RPD) for individual PAHs and/or total PAHs and PAH pattern evaluation will be used to compare data. The specific statistical tests and acceptance criteria will be determined through discussion with the Trustees and BP.
- Acquire quantitative results for alkylated PAHs (Table 1.1a of AQAP), saturated hydrocarbons/ total extractable hydrocarbons, (Table 1.1b of AQAP), and petroleum biomarkers (Table 1.1e of AQAP).
- Acquire percent moisture and Total Organic Carbon (TOC) data for each sediment/soil sample listed in Table 3.

4.0 Level of Effort and Logistics

Once all parties agree on the list of samples for analysis and experimental design, Alpha will be given the authorization to proceed with analysis. Any issues encountered by Alpha during sample processing (e.g., problems in extraction or analysis), will be communicated to William B. Driskell who will be responsible for informing all parties of the issues and resolutions or modifications to the addendum plan.

The lab-verified results will be provided simultaneously to the Parties for distribution to the Trustees and BP. Results will be reviewed and discussed through the Chemistry TWG.

5.0 Milestones and Deliverables:

5.1 Sample Analysis:

- Samples will be analyzed with a 28-day turn-around-time (TAT)

5.2 Reporting:

The following items will be provided simultaneously as deliverables to the Trustees and BP NRDA. Delivery will be by e-mails from Alpha to the recipients listed below when feasible, and in all cases to an ftp site to be established by Cardno ENTRIX for direct access to laboratory records by all plan principals:

  - On behalf of the Trustees: William B. Driskell (bdriskell@comcast.net)
  - On behalf of Louisiana Trustees: Amanda Vincent (amanda.vincent@la.gov)
  - On behalf of BP NRDA: Dreas Nielsen (dnielsen@integral-corp.com)
- Electronic data deliverable (EDD) in NOAA QueryManager format
- Excel spreadsheet in standard Alpha format with histogram plots and quantitative results for samples, quality control sample, and SRMs.
- Level IV data package for quantitative analysis of PAH, Biomarkers, Saturated Hydrocarbons, and % moisture.
- Photographs of all samples and sample jars
- All laboratory notes regarding handling and homogenization of replicates
- The data released by the laboratory will be available for technical evaluation by all plan principals while they are undergoing validation as cooperative data by 3rd party validators for the Trustees and BP NRDA (EcoChem and Cardno ENTRIX). There shall be no restriction on use of the data for the holding time study as released by the laboratory, recognizing that the final data for public release may be subject to qualification as appropriate.

All documentation associated with analyzing the samples will be preserved in accordance with the legal documentation preservation order.

5.3 Data Sharing:

Alpha shall simultaneously deliver laboratory-verified data, including all necessary metadata, generated as part of this addendum work plan as a Laboratory Analytical Data Package (LADP) to EcoChem, Inc. on behalf of the Trustees, the Louisiana Oil Spill Coordinator's Office (LOSCO) on behalf of the State of Louisiana and to BP (or Cardno ENTRIX on behalf of BP).

Upon completion of all analyses, the final Alpha data report and electronic files along with the results of the original analyses performed on the same samples will be validated and made available to all Parties.

6.0 Key Personnel:

• Study Director	Dennis Beckmann / BP
• Analytical Leaders	Robert Barrick (Cardno ENTRIX on behalf of BP) and Greg Baker (NOAA on behalf of the Trustees)
• QA Manager (Trustees)	Ann Bailey (EcoChem on behalf of the Trustees) and Cheryl Randle (Cardno ENTRIX on behalf of BP)
• Data Analyst (Trustees)	Linda Cook (Exponent on behalf of BP) and William Driskell (PECI on behalf of the Trustees)

7.0 Safety Plans:

Health and safety protocols in accordance with good laboratory practice will be followed by Alpha, who is responsible for developing and implementing requirements.

8.0 References

NOAA. 2012. Analytical Quality Assurance Plan, version 3.1. Mississippi Canyon 252 (Deepwater Horizon) Natural Resource Damage Assessment. Prepared by EcoChem. November 15, 2012.

NOAA. 2011. NRDA Laboratory Plan, version 2.0. Holding Time Study for Environmental Samples in Frozen Archives: Laboratory Analysis Plan. August 18, 2011.

9.0 Costs

All laboratory costs will be billed as is done with other cooperative laboratory analyses at Alpha Analytical Laboratory through Industrial Economics, Incorporated (IEc) under their contract for Deepwater Horizon Oil Spill Natural Resource Damages Assessment Services.

Table 3. Proposed List of Samples for Evaluation

Study	Client ID	Lab ID	Matrix	TPAH50 (µg/Kg) [1]	Date Collected	Date Analyzed	Latitude	Longitude
Nearshore Sed & Water--Preassessment-Early OCT 2010	LAAQ43-A1013-SC714	1010201-06	Sediment	780	10/13/2010	10/27/2010	29.19468	-89.03949
Oyster--Preassessment-Oyster Sampling--2010	LAAQ39-A0907-SH9001	1009142-01	Sediment	440	9/7/2010	10/14/2010	29.27703	-89.94175
Nearshore Sed & Water--Preassessment-Late AUG 2010	LAAP39-A0824-SC504	1008350-10	Sediment	1900	8/24/2010	9/5/2010	29.44769	-89.92625
Nearshore Sed & Water--Preassessment-Late AUG 2010	LAAP39-A0824-SC508	1008350-12	Sediment	1200	8/24/2010	9/5/2010	29.44757	-89.92661
Shoreline-Coastal Wetland Vegetation Plan--2010	LAAP39-A1023-SN6703_COMP	1104239-12	Soil	210000	10/23/2010	7/16/2011	29.45647	-89.88701
MDEQ Preassessment Early MAY 2010	MS047-SMS05-050310-01-S	1103015-07	Sediment	240	5/03/2010	4/12/2011	30.355139	-88.885861

Notes:

[1] TPAH50 is the sum of Alkylated PAHs listed in Table 1.1a of the AQAP excluding Retene and Perylene. Non-detected results were assumed to have a value of zero ("0") in the summation.